Army Research Laboratory

Yttria Nanoparticle Reinforced Commercially Pure (CP) Titanium

by Sesh Tamirisa

ARL-CR-0680 September 2011

Prepared by

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Under Contract

W911NF-11-2-0003

ERRATA SHEET

re: ARL-CR-0680, *Yttria Nanoparticle Reinforced Commercially Pure (CP) Titanium*, March 2012, by Sesh Tamirisa

This is an errata sheet for ARL-CR-0680. Please replace the old version of the report with the new version, and destroy the old version.

Page	Should Read
The tensile data were switched for the CP Ti and CP Ti + 0.3 Y_2O_3 samples in table 1. These changes in the data set have affected the text of the report in the following sections.	
ii	Adjusted abstract.
1	Adjusted summary.
6	Adjusted paragraph.
7	Corrected table 1.
7	Corrected table 1.
8	Corrected figure 4.
8	Corrected figure 5.
10	Corrected figure 7.
11	Corrected figure 8.
11	Corrected figure 9.
12	Adjusted conclusion.
12	Adjusted future work.

Vincent H. Hammond

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ARL-CR-0680 **September 2011**

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14. ABSTRACT

Previous literature reports have indicated as much as an approximately twofold increase in tensile properties in commercially pure (CP) titanium (Ti) following the introduction of yttria (Y_2O_3) nanoparticles. However, these initial studies used laboratory-scale samples produced via an arc-melting process. In order to evaluate the potential for scale-up, CP Ti powders containing both yttria nanoparticles as well as titanium boride (TiB) reinforcements were produced through gas atomization. After consolidation and extrusion, room temperature tensile tests were conducted to determine the influence of reinforcements on strength and ductility. Three alloy powders—CP Ti, CP Ti + 0.3% Y_2O_3, and CP Ti + 0.3% Y_2O_3 + 0.5 B—were fabricated using a conventional Ti powder metallurgy route. Powder compacts were fabricated via hot isostatic pressing and billets were extruded to produce 12.7-mm-diameter bars. Room temperature testing indicated that the addition of 0.3% Y_2O_3 reduced the tensile strength by approximately 20%. The addition of both 0.5% B and 0.3% Y_2O_3 increased the tensile yield strength by 33% relative to CP Ti. The addition of 0.5% B increased the tensile modulus of CP Ti by 20%.

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Summary

A previous report in the literature[1] asserted that the doubling of tensile properties is achievable via nano-sized yttria (Y_2O_3) dispersion in titanium (Ti). These initial studies reported proof of concept using small laboratory-scale samples produced via an arc-melting process, but follow-on studies to establish the technology and applications have been lacking. The objective of this work is to study the scale-up feasibility of yttria dispersed Ti and investigate the influence of yttria nanoparticles on the tensile properties of Ti. The focus of this effort is to perform initial experiments to determine if this strengthening effect occurs in metal powder produced through gas atomization. In addition, a higher strength version of the material was sought through the addition of boron (B). This project is designed to explore the efficiency of nano-sized yttria at improving tensile properties of commercially pure (CP) Ti for U.S. Army applications. This phase of the project is focused on evaluating the tensile properties of a limited number of samples with specified compositions.

CP Ti and inert gas atomization were selected as material and process in this study. Scale-up feasibility of nano-yttria dispersed CP Ti was successfully demonstrated by producing large quantities of CP Ti alloys via conventional titanium powder metallurgy route. Three alloys—CP Ti, CP Ti + 0.3% Y_2O_3, and CP Ti + 0.3% Y_2O_3 + 0.5 B—were made. Powder compacts were fabricated via hot isostatic pressing and billets were extruded to produce 0.5 in (12.7 mm) diameter bars. Room temperature tensile testing was performed on multiple specimens machined from bars. Addition of 0.3% Y_2O_3 reduced the tensile strength of CP Ti by approximately 20%. The tensile yield strength of the CP Ti + 0.3% Y_2O_3 +0.5% B alloy was 33% higher relative to CP Ti and 66% relative to the CP Ti + 0.3% Y_2O_3 alloy. The addition of 0.5% B increased the tensile modulus of CP Ti by 20% in addition to significantly increasing strength while maintaining adequate ductility. Yttria addition, on the other hand, resulted in only a modest increase in total elongation.

The feasibility of producing yttria-dispersed Ti and the resultant tensile properties were successfully demonstrated. In contrast to the previous literature report, the yttria-reinforced alloy actually demonstrated reduced tensile strengths. In contrast, the boron-containing alloy demonstrated significant improvements in tensile strengths relative to CP Ti and the CP Ti/yttria alloy. The use of TiB reinforcements represents one method for producing higher strength CP Ti alloys with the potential for significant weight reduction benefits for U.S. Army applications.

[1] de Castro, V.; Leguey, T.; Munoz, A.; Monge, M. A.; Pareja, R. Microstructure and tensile properties of Y_2O_3-dispersed titanium produced by arc melting. *Materials Science and Engineering A* **April 2006**, *A422* (1–2), 189–197.

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1. Fabrication of Yttria Nano-particle Reinforced CP Titanium

Pre-alloyed powders of commercial purity (CP) titanium (Ti), CP Ti + 0.3% yttria (Y_2O_3), and CP Ti + 0.3% yttria + 0.5% boron (B) were produced via inert gas atomization technique. As-atomized powders were sieved to obtain –35 mesh (500-µm-mesh opening size) fractions. Chemical analysis (wt.%) and sieve analysis of all three powder compositions are given in figure 1. In comparison to typical CP Ti Grade 2 chemistry (0.3% max iron [Fe] and 0.25% max oxygen [O]), the Fe and O contents were lower in the powder materials produced in this study. However, these levels were similar in the baseline CP Ti and yttria-modified CP Ti, which allows accurate comparison of properties.

ATI Powder Metals

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Page 1 of 1

Date: February 25, 2011

CERTIFICATE OF TEST

CR Order: T802

Customer:	Purchase Order No:	Purchase Order Date:
FMW Composite Systems	11462	December 7. 2010

Specification:

Item No.:	Weight:	Product Description:
1	15.0 lbs	CP Ti Grade 2 Powder, -35 Mesh, Heat 4255
2	65.0 lbs	CP Ti Grade 2 + $0.45Y_2O_3$ Powder, -35 Mesh, Blend B630
3	65.0 lbs	CP Ti Grade 2 + $0.45Y_2O_3$,+ 0.5B Powder, -35 Mesh, Blend B631

Chemical Analysis (Wt%):

Lot	Fe	Y	B	C	O	N	H	Ti
4255	0.038	0.005	----	0.051	0.088	0.006	0.003	Bal.
B630	0.044	0.30	----	0.035	0.107	0.003	0.003	Bal.
B631	0.053	0.32	0.53	0.041	0.101	0.005	0.004	Bal.

Powder Sieve Analysis (% Under):

Lot	-35	-45	-60	-80	-100	-140	-200	-230	-270	-325	Tap Density (g/cc)	Flow Rate (sec)
4255	100.0	91.1	72.4	51.6	41.0	19.1	7.1	3.8	2.4	1.5	2.53	38
B630	100.0	88.4	65.5	46.0	38.2	21.4	11.2	7.2	5.0	3.3	2.91	33
B631	100.0	87.9	65.9	46.3	37.9	20.2	10.3	6.3	4.6	3.0	2.84	31

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Figure 1. Chemical analysis and sieve analysis of yttria nanoparticle reinforced CP Ti powders.

2. Tensile Properties of Yttria Nano-particle Reinforced CP Titanium

Powders of about 10 lb from each composition were packed in mild steel cans, hot offgassed at 500 °F for 2 h, and vacuum sealed. Sealed cans were subjected to hot isostatic pressing (HIP) at 1750 °F and 15 ksi for 3 h, and fully dense powder compacts were produced. Compacts were decanned and machined into billets of 2.95 inches in diameter and 7 inches in height. Billets were extruded into 0.75-in-diameter bars using conditions of 1650 °F billet temperature with 1 h soak time, 12:1 extrusion ratio, 100 in/min ram speed, and air cooling after extrusion. Pictures of powder cans, machined billets, and extruded bars are shown in figure 2.

Figure 2. Fabrication process flow of yttria nanoparticle reinforced CP Ti extruded bars.

Blanks of 3-in length were cut from extrusions and tensile specimens of 0.25-in gauge diameter and 1-in gauge length were machined. Six samples from each alloy were machined to tensile specimen drawing shown in figure 3. Tensile testing of specimens was performed at room temperature per the American Society for Testing and Materials (ASTM) standard E8 using an initial ram speed of 0.005 in/in/min (0.00212 mm/mm/s). Digital elongation data were recorded using an extensometer of 1-in length attached to the gauge portion of the specimen.

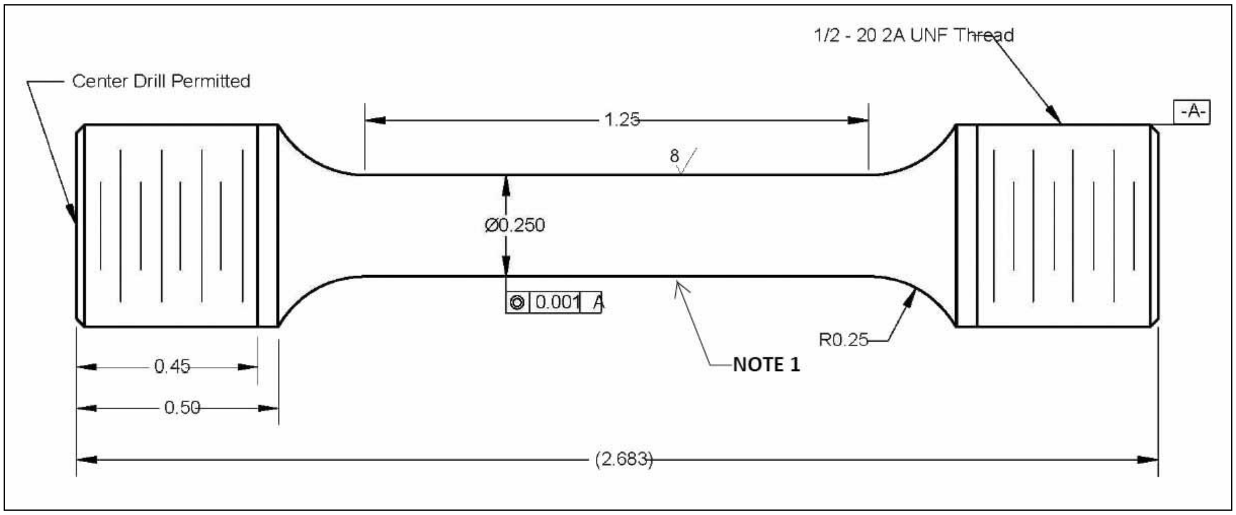

Figure 3. Tensile specimen drawing used for evaluating CP Ti extruded bars.

Tensile data generated on all three alloys are presented in table 1. Tensile stress-strain curves of all tested samples are presented in figures 4–6 (the data on CP Ti and CP Ti + 0.3% Y_2O_3 samples were truncated as the number of data points exceeded the Excel plotting limit). Tensile strengths (0.2% yield and ultimate) of three alloys are compared in figure 7. The addition of 0.3% Y_2O_3 reduced the tensile strength of CP Ti by 20% (–50 MPa in tensile yield strength [TYS] and –80 MPa in ultimate tensile strength [UTS]) while the tensile elongation value remained unchanged. For both alloys, it was noted that tensile strengths were significantly lower and the tensile elongations were higher compared to typical CP Ti Grade 2 (275 MPa guaranteed minimum TYS and 20% tensile elongation). These results are consistent with the lower Fe and O contents in powder materials. The addition of 0.5% B increased the tensile strength (22–33%, +80 MPa in TYS and +90 MPa in UTS) compared to the baseline CP Ti. The addition of B also increased the tensile modulus by 20% (+20 GPa) compared to the baseline CP Ti and yttria-modified CP Ti, as shown in figure 8. In the B-modified CP Ti, a reduction in total tensile elongation to 28% occurred compared to the baseline alloy (figure 9). However, the tensile elongation is well above the desired value of 10–15% for a majority of applications.

Table 1. Room temperature tensile test data on yttria nanoparticle reinforced CP Ti bars.

Alloy	ID	Dia., mm	TYS		UTS		TE	TM	
			MPa	ksi	MPa	ksi	%	GPa	Msi
CP Ti	#1-1	6.345	240.9	35	407.4	59.1	43.0	103.8	15.1
	#1-2	6.317	245	36	409.7	59.4	43.5	105.7	15.3
	#1-3	6.325	247.9	36	410.5	59.5	44.0	109.2	15.8
	#1-4	6.322	248	36	409.7	59.4	49.6	103.8	15.1
	#1-5	6.317	249	36	408.3	59.2	44.3	112.8	16.4
	#1-6	6.314	251	36	412.9	59.9	46.8	106.6	15.5
		Avg	**247**	**36**	**410**	**59**	**45**	**107.0**	**15.5**
		Std Dev	3.6	0.5	1.9	0.3	2.5	3.5	0.5
CP Ti + 0.3 Y_2O_3	#2-1	6.320	210.0	31	326.2	47.3	54.4	111.8	16.2
	#2-2	6.322	198	29	323.9	47.0	47.8	103.2	15.0
	#2-3	6.327	197.1	29	329.8	47.8	49.2	105.8	15.3
	#2-4	6.335	193	28	331.6	48.1	47.4	106.8	15.5
	#2-5	6.340	192	28	333.0	48.3	44.5	111.4	16.2
	#2-6	6.325	184	27	334.3	48.5	53.1	97.5	14.1
		Avg	**196**	**28**	**330**	**48**	**49**	**106.1**	**15.4**
		Std Dev	8.6	1.2	4.0	0.6	3.7	5.4	0.8
CP Ti + 0.3 Y_2O_3 + 0.5 B	#3-1	6.335	316	46	488.0	70.8	28.7	125.4	18.2
	#3-2	6.320	332	48	495.7	71.9	27.0	128.5	18.6
	#3-3	6.345	331	48	503.0	73.0	27.9	137.7	20.0
	#3-4	6.335	328	48	505.2	73.3	28.0	114.5	16.6
	#3-5	6.342	324	47	502.2	72.8	29.4	121.5	17.6
	#3-6	6.330	328	48	503.1	73.0	27.3	134.5	19.5
		Avg	**327**	**47**	**500**	**72**	**28**	**127.0**	**18.4**
		Std Dev	5.9	0.8	6.5	0.9	0.9	8.5	1.2

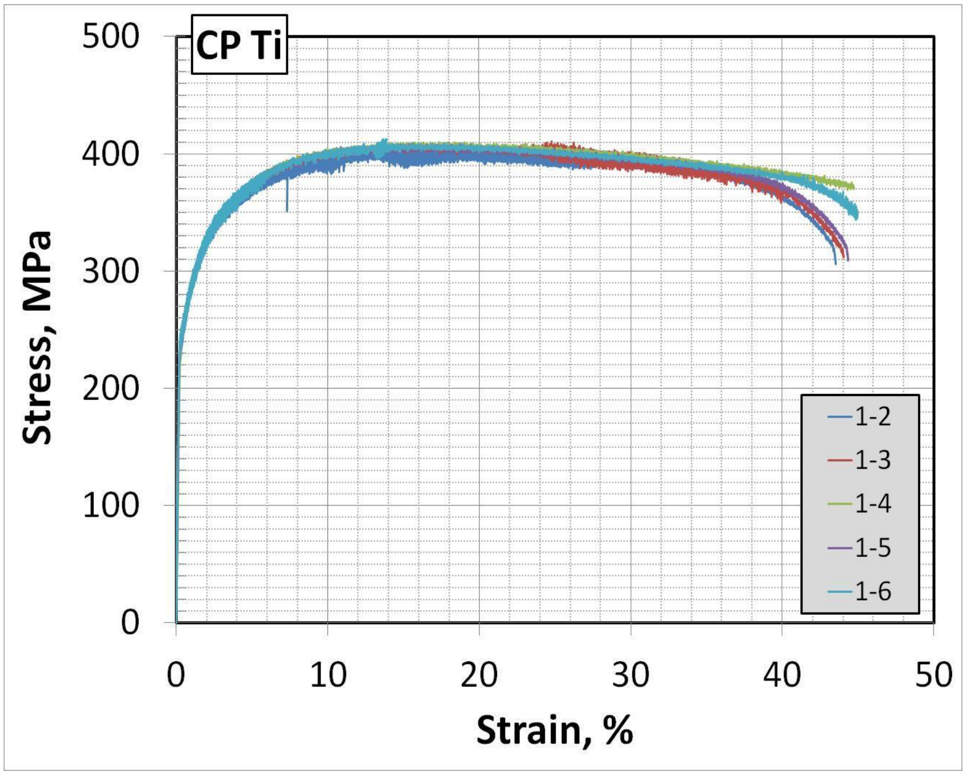

Figure 4. Tensile stress-strain curves at room temperature on CP Ti extruded bar.

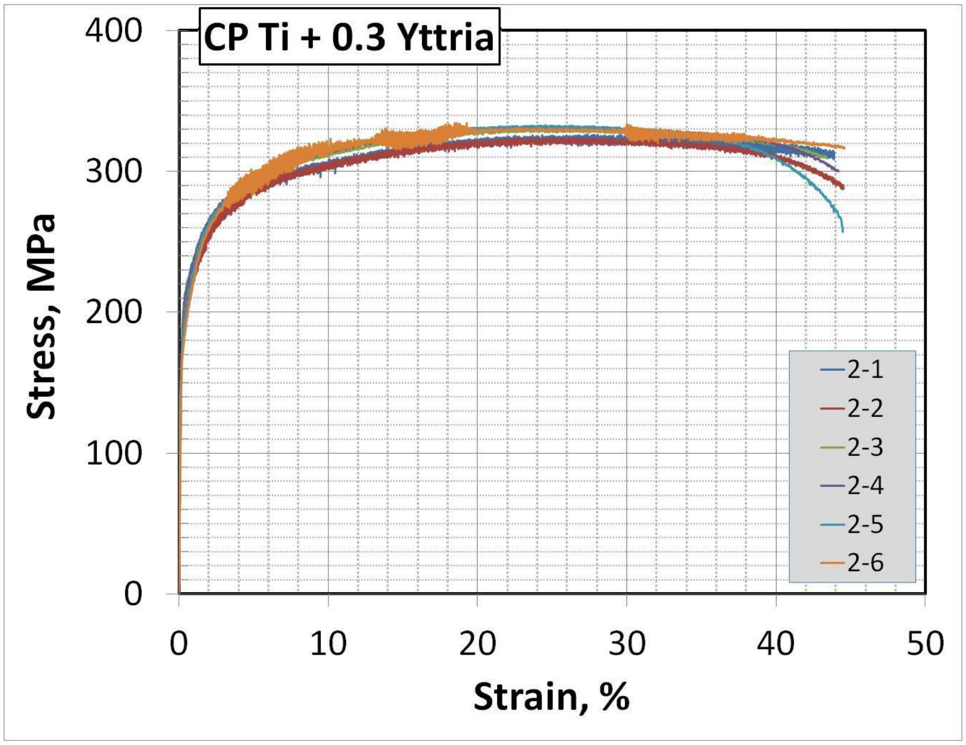

Figure 5. Tensile stress-strain curves at room temperature on CP Ti + 0.3 yttria extruded bar.

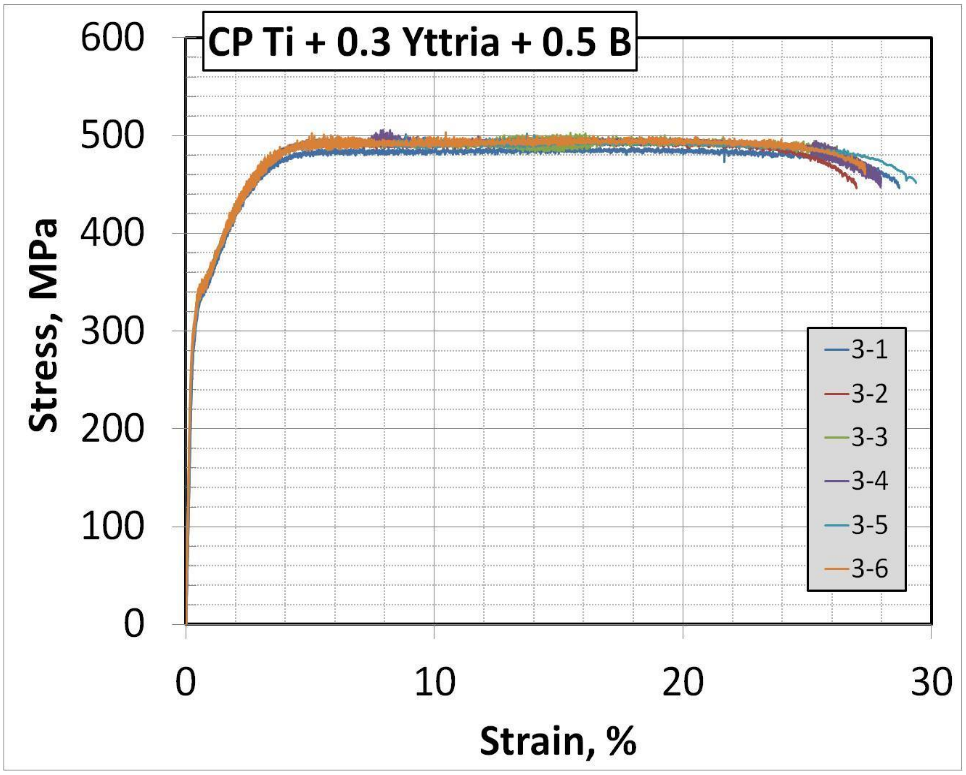

Figure 6. Tensile stress-strain curves at room temperature on CP Ti + 0.3 yttria + 0.5 B extruded bar.

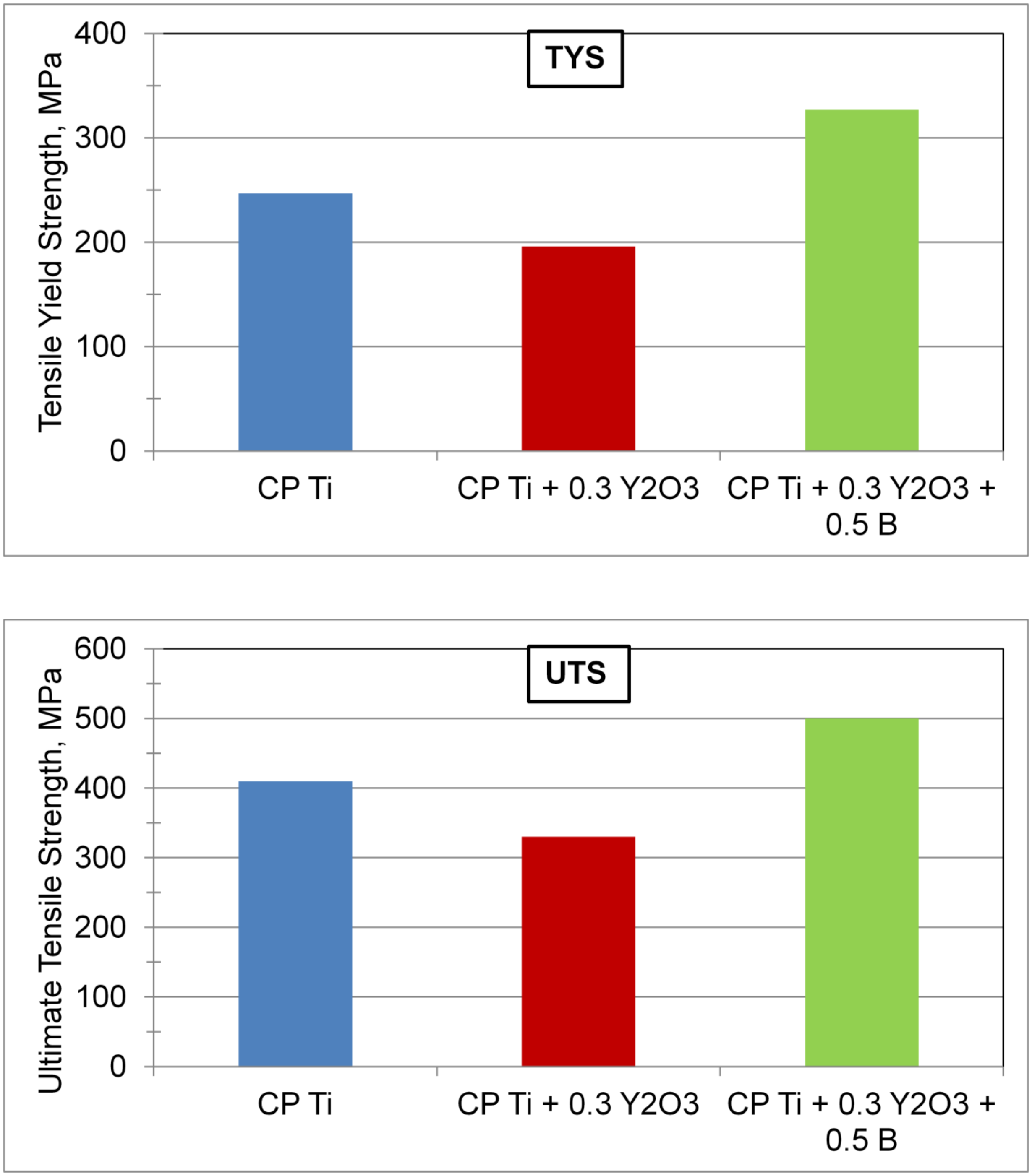

Figure 7. Comparison of tensile strengths (average values) of nano-yttria particle reinforced CP Ti extrusions.

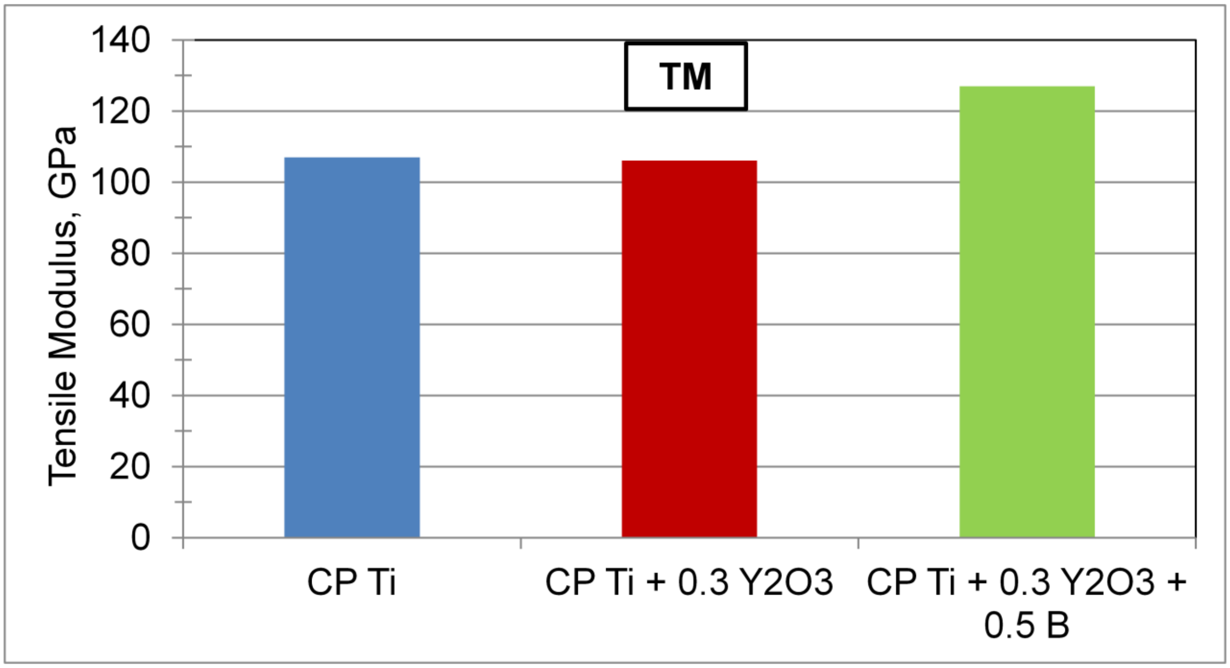

Figure 8. Comparison of tensile modulus (average values) of nano-yttria particle reinforced CP Ti extrusions.

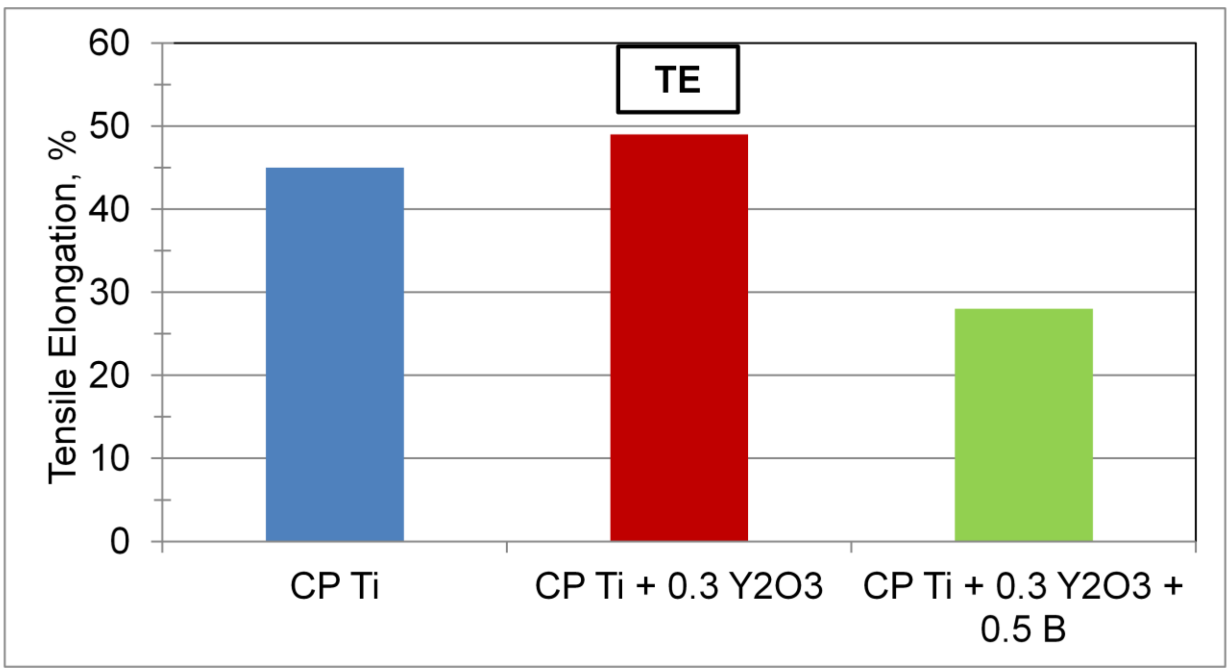

Figure 9. Comparison of tensile elongation (average values) of nano-yttria particle reinforced CP Ti extrusions.

3. Conclusions

The following is a summary of the results of this study:

- The scale-up feasibility of nano-yttria dispersed CP Ti was successfully demonstrated by producing large quantities of modified alloys using conventional Ti powder metallurgy production processes.
- The addition of 0.3% Y_2O_3 reduced the tensile strength of CP Ti by approximately 20%.
- The TYS of the CP Ti + 0.3% Y_2O_3 + 0.5% B alloy was 33% higher than the CP Ti alloy, while the UTS was increased by 22%. In comparison to the CP Ti + 0.3% Y_2O_3 alloy, the yield and ultimate strengths were increased 66% and 51%, respectively.
- Addition of 0.5% B increased the tensile modulus of CP Ti by 20% in addition to significantly increasing strength while maintaining adequate tensile elongation. Yttria addition, on the other hand, provided only an increase in total elongation to CP Ti.

4. Future Work

The following future work needs to be conducted to further this effort:

- We need to determine the reason for the reduction in tensile strengths observed for the CP Ti + 0.3% Y_2O_3 alloy.
- Optimization of the alloy chemistry to match the Fe and O contents to those in typical CP Ti Grade 2 could provide further improvements in tensile strength.
- Optimization of the B content is necessary to obtain optimal property combinations in CP Ti.
- Microstructural evaluations need to be conducted to understand the structure-property relationships and guide microstructural engineering.

List of Symbols, Abbreviations, and Acronyms

ASTM	American Society for Testing and Materials
B	boron
CP	commercially pure
FE	iron
O	oxygen
Ti	titanium
TiB	titanium boride
TYS	tensile yield strength
UTS	ultimate tensile strength
wt%	weight percent
Y_2O_3	yttria

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